

High-strength Carbon Nanotube/Polyetheretherketone Composite Films with Tunable Impregnation for Electromagnetic Interference Shielding and Thermal Management in Harsh Environments

Chun-Qi Guo, Yu Bai, Liu-Bo Liang, Shu-Shu Jia, Xin-Yuan Shi, Gui-Bin Wang, and Shu-Ling Zhang*

Key Laboratory of High Performance Plastics, Ministry of Education, College of Chemistry, Jilin University, Changchun 130012, China

Electronic Supplementary Information

Abstract With the rapid development of lightweighting and information technology, there is an urgent need for lightweight, high-strength, and multifunctional polymer-based composite films that integrate electromagnetic interference (EMI) shielding and thermal management capabilities. However, maintaining strength and functional stability in harsh environments still faces significant challenges. A carbon nanotube/polyetheretherketone (CNT/PEEK) composite film was fabricated *via* vacuum-assisted impregnation and wet-pressing techniques. By adjusting the molecular weight of the PEEK matrix, the composite film attained a high tensile strength of 218.42 MPa. This study elucidates a dual-competition mechanism during the impregnation of polymer solutions with varying molecular weights. The highly conductive carbon nanotube (CNT) film, serving as the functional filler, endows the CNT/PEEK composite film with high EMI shielding effectiveness (SE_T of 47 dB at 10 μm , specific shielding effectiveness per thickness (SE/t) of 4700 dB/mm) and outstanding dual-conversion capabilities (electrothermal: 3 V, 79 °C; photothermal: 2.0 W/cm², 178 °C). Furthermore, the semi-crystalline nature of PEEK ensures that the composite film maintains excellent stability even after undergoing high/low-temperature impacts and long-term erosion under acid/alkali corrosive conditions. The high-strength CNT/PEEK composite film shows promise for applications, such as EMI shielding and thermal management in harsh environments.

Keywords Molecular weight regulation; High strength; Electromagnetic interference (EMI) shielding; Thermal management; Performance stability

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INTRODUCTION

While electromagnetic waves drive the development of the information society, their extensive radiation also brings the dual challenges of electromagnetic interference (EMI) to equipment and excessive exposure of humans. This has spurred the exploration of advanced EMI shielding materials.^[1–4] Traditional metallic shielding materials are limited by their high densities, processing difficulties, and susceptibility to corrosion, making them unsuitable for lightweight portable devices and harsh environments.^[5–8] In contrast, polymer-based EMI materials have been widely applied because of their low density, easy processing, and corrosion resistance.^[9–13]

Polymer-based EMI shielding composite films have attracted significant attention because of their light weight and processability.^[14–20] Additionally, to further enhance the processing performance and molding capability of the composite film, it is also necessary to have good flexibility.^[21,22] Conduc-

tive networks within polymer matrices can achieve a high EMI SE.^[23–25] Lei *et al.* constructed an efficient conductive network by depositing a silver fractal dendrite (AgFD) layer on a poly(vinylidene fluoride) (PVDF) film, and prepared an AgFD/PVDF composite film, which achieved 46.8 dB in the X-band.^[26] Guo *et al.* prepared a mechanically robust thermoplastic polyurethane/graphene nanosheet (TPU/GN) Janus film through chemical stripping and evaporation processes, which exhibited a tensile strength of 26.2 MPa and EMI SE of 49.1 dB in the X-band.^[27] Su *et al.* developed graphene nanosheet/aramid nanofiber (GNS/ANF) nanocomposite films *via* a sol-gel strategy, achieving an EMI SE of 31 dB and tensile strength of 131.17 MPa.^[28] Despite these advances, limited attention has been paid to maintaining performance stability in harsh environments, which poses challenges for practical applications.

Polyetheretherketone (PEEK) is the preferred resin because of its high strength, excellent thermal stability, and corrosion resistance.^[29] Carbon nanotubes (CNTs) possess outstanding electrical conductivity and remarkable light absorption capability. Therefore, the introduction of CNTs as functional fillers

* Corresponding author, E-mail: zsl@jlu.edu.cn

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can endow the PEEK-based composites with excellent EMI shielding, electrothermal conversion, and photothermal conversion properties.^[30,31] Such multifunctional conductive polymer composites have attracted increasing attention for use in next-generation intelligent devices and harsh environment applications.^[32,33] In previous work, we fabricated sandwich-structured PEEK/carbon nanotube (CNT) composite films with a total thickness of 160 μm , tensile strength of 78 MPa and EMI SE of 18.7 dB.^[34] To further enhance the mechanical strength and EMI SE of the PEEK-based composite films, a 36 μm thick EMI shielding composite film was subsequently prepared using phenylethynyl-rafted CNT film as the functional filler and phenylethynyl-terminated PEEK as the resin matrix. This composite film exhibited a tensile strength of 257 MPa and EMI SE of 52 dB within the frequency range of 8.2–12.4 GHz.^[35] To further improve the EMI SE without significantly compromising the mechanical performance, a 43 μm thick composite film was fabricated by incorporating both phenylethynyl-grafted CNTs and MXene as functional fillers into a phenylethynyl-terminated PEEK matrix. The final composite achieved a tensile strength of 233 MPa and EMI SE of 73 dB in the X-band.^[36] These studies demonstrate the potential of phenylethynyl modification and crosslinking. However, the influence of the molecular weight of the resin on the properties of the composite film remains poorly understood, particularly in terms of its influence on the impregnation behavior and multifunctional performance.

In this study, CNT/PEEK composite films were fabricated by systematically controlling the molecular weight of the resin and employing wet-pressing technology. By designing a soluble and amorphous An-PEEK precursor for crystalline PEEK resin, solution processing under mild conditions was successfully achieved, thereby overcoming the limitations of difficult solution processing of crystalline PEEK. Through the systematic modulation of the molecular weight of the PEEK matrix, a dual competition mechanism governing the impregnation behavior of polymer solutions with varying molecular weights was elucidated. At a critical molecular weight of 4.75×10^4 g/mol ($[\eta] \approx 0.541$ dL/g), adequate fluidity for near-complete impregnation and ample chain entanglement yielded a composite film with a high tensile strength of 218.42 MPa. The highly conductive CNT film, serving as the functional filler, endowed the CNT/PEEK composite film with excellent EMI shielding performance (SE_T of 47 dB at 10 μm ,

SE/t of 4700 dB/mm) and outstanding dual-conversion capabilities (electrothermal: 3 V, 79 $^{\circ}\text{C}$; photothermal: 2.0 W/cm², 178 $^{\circ}\text{C}$). Furthermore, the semi-crystalline nature of PEEK ensured that the composite film maintained excellent stability even after undergoing high/low-temperature impacts and long-term erosion under acid/alkali corrosive conditions. This work provides a molecular-weight-governed design strategy for high-strength composites without a crosslinking reaction, resulting in a high-strength CNT/PEEK composite film that is promising for EMI shielding and thermal management in harsh environments.

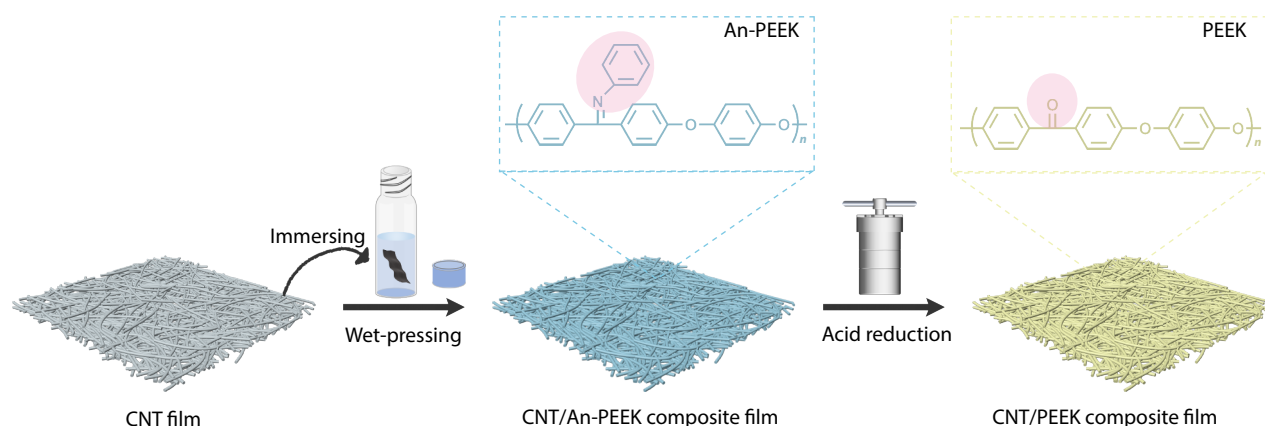
EXPERIMENTAL

Fig. 1 illustrates the preparation procedure of the CNT/PEEK composite films. The relevant section information is presented in the electronic supplementary information (ESI).

RESULTS AND DISCUSSION

Preparation and Characterization of An-PEEK Precursors

The chemical structure of *N*-phenyl-1,1-bis(4-fluorophenyl) methanimine was verified by ¹H-NMR spectroscopy (Fig. S4 in ESI). An-PEEK was used as an example for ¹H-NMR characterization to demonstrate the feasibility of the above synthesis method, as shown in Fig. 2(A). The cleavage patterns of the proton signals on the benzene ring were aligned with theoretical predictions, confirming the successful synthesis of the monomer and An-PEEK. Fig. 2(B) shows the FTIR spectra of An-PEEK measured both before and after acid reduction, clearly demonstrating the reduction reaction. The band at 1590 cm⁻¹ corresponds to C=N stretching, while that at 1303 cm⁻¹ arises from C—N stretching. Following the acid reduction treatment, the C—N band disappeared and a new C=O stretching band appeared at 1650 cm⁻¹. This spectral evolution demonstrates the conversion of the ketimine structure in An-PEEK to the ketone carbonyl structure, which is characteristic of PEEK. Additional bands at 1224 cm⁻¹ (C—O of aryl ether) and 1495 cm⁻¹ (C=C of benzene ring) matched typical PEEK signatures, further verifying successful reduction. As shown in Fig. 2(C) and Fig. S5 (in ESI), the XRD patterns of An-PEEK and PEEK reveal distinct structural differences. An-PEEK exhibited a single broad peak at 18.53 $^{\circ}$, which is characteristic of an amorphous polymer.



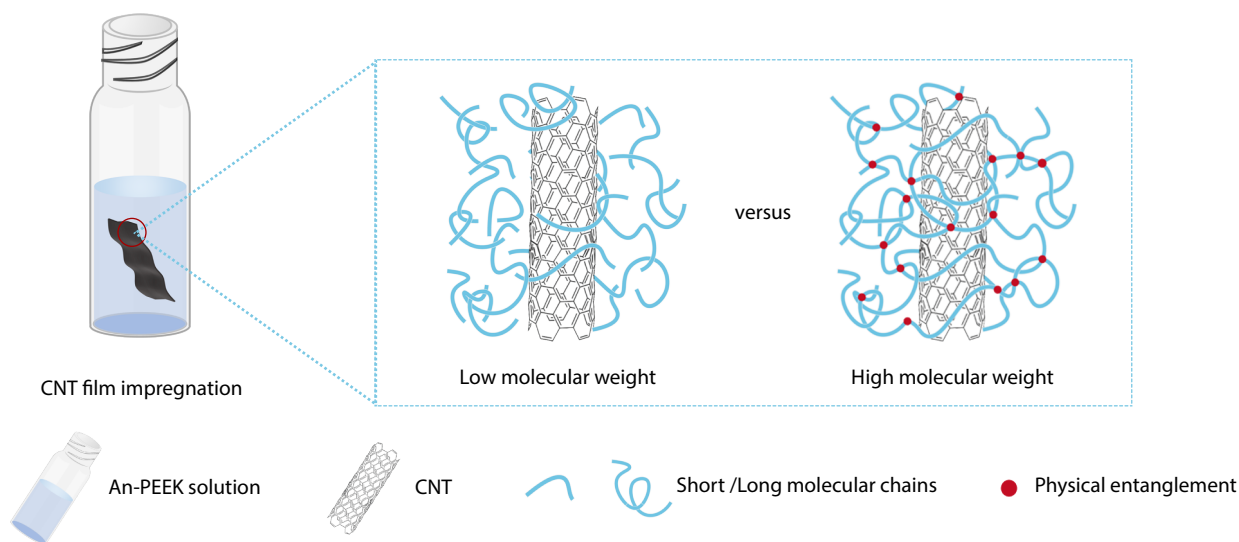


Fig. 1 Schematic illustration of the fabrication process of CNT/PEEK composite film.

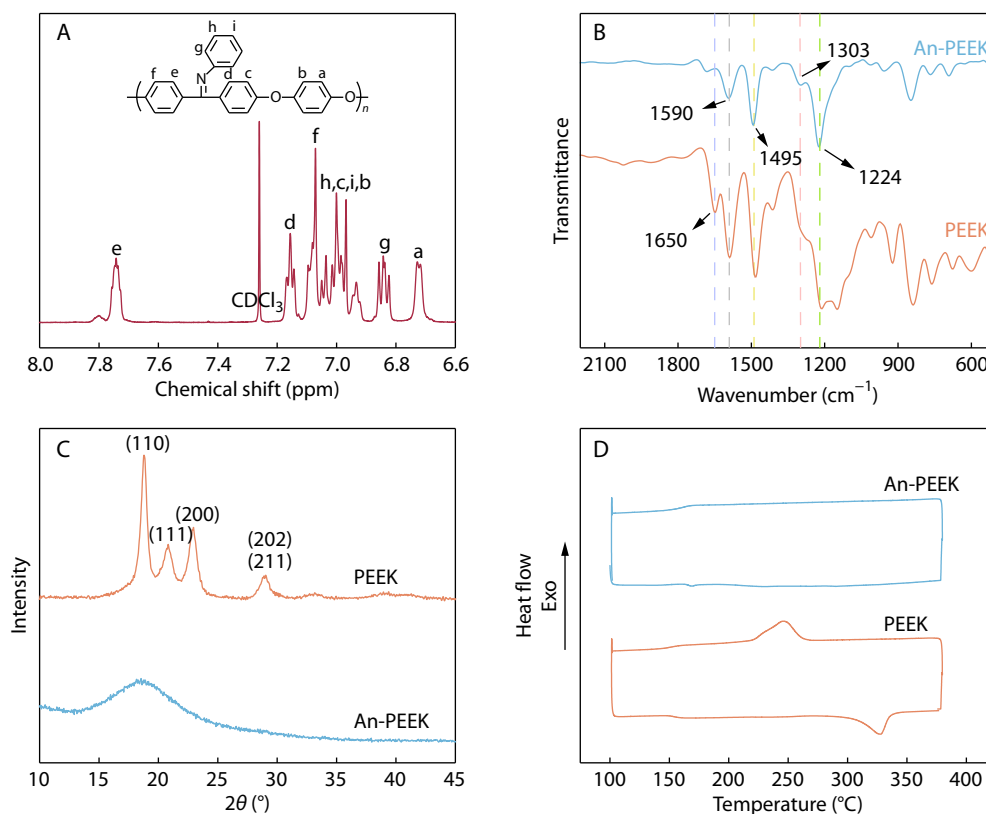


Fig. 2 Characterization of the An-PEEK and PEEK. (A) ^1H -NMR spectrum of An-PEEK; (B) FTIR spectra, (C) XRD patterns and (D) DSC curves of An-PEEK and PEEK.

In contrast, PEEK displayed four primary crystalline diffraction peaks: (110) at 18.96° , (111) at 20.98° , (200) at 23.09° , and overlapping (211)/(202) peaks at 29.04° . These results demonstrated that the reduction treatment of An-PEEK regenerated the carbonyl group, enhancing the polymer chain regularity and conferring crystallinity.^[37] The polymers before and after acid reduction were characterized by DSC. As shown in Fig. 2(D), the DSC curve of amorphous An-PEEK exhibited neither a melting peak

nor a crystallization peak. This absence of crystallization behavior stems from the ketimine structure, which suppresses the crystallization.^[38] Following acid reduction, the DSC curve of the resulting PEEK displayed both a crystallization peak and a melting peak (the corresponding data are summarized in Table S2 in ESI). The enthalpy of the melting peak exhibited a decreasing trend with increasing molecular weight. This behavior is attributed to the dependence of polymer crystallization on chain

diffusion and structural reorganization. In the case of lower-molecular-weight polymers under identical conditions, their shorter chain lengths and lower viscosities lead to enhanced molecular mobility and more effective chain rearrangement, ultimately resulting in higher crystallinity.^[39–41]

Effect of An-PEEK Molecular Weight on Solution Properties and Impregnation Behavior

Figs. 3(a)–3(e) show the surface morphology of CNT/An-PEEK-5 composite films with different mass concentrations (0 wt%, 2 wt%, 4 wt%, 8 wt%, 12 wt%). The An-PEEK resin is uniformly combined with the CNT bundles, resulting in a smooth surface of the film. The pristine CNT film exhibits randomly oriented nanotubes and contains numerous voids. At a lower An-PEEK concentration (2 wt%), insufficient resin fails to fill the voids (Fig. 3b), resulting in numerous residual pores within the film. These pores significantly compromise load transfer efficiency, thereby diminishing the film's mechanical properties. Conversely, at a higher An-PEEK concentration (12 wt%), the voids are completely filled; however, excess resin forms distinct additional layers (Fig. 3e), which can adversely affect the mechanical properties. Fig. 3(f) displays the EDS elemental mapping of carbon and oxygen on the surface of the pristine CNT film (Fig. 3f-a1) and the CNT/PEEK-5 composite film (Fig. 3f-e1). The pristine film displays negligible oxygen content, while the composite film features a markedly higher surface oxygen concentration. As presented in Fig. 3(g), the FTIR spectra correspond to CNT/An-PEEK

and CNT/PEEK. The absorption band at 1590 cm^{-1} corresponds to the C=N stretching vibration of An-PEEK, while the band at 1303 cm^{-1} corresponds to the C—N stretching mode. After acid reduction, the C—N stretching signal disappears, and a new absorption band at 1650 cm^{-1} emerges, assigned to the C=O stretching vibration. This indicates that acid reduction converts the CNT/An-PEEK to CNT/PEEK composite film. The CNT and An-PEEK content within composite films fabricated by varying An-PEEK concentration are shown in Fig. 3(h). The CNT content in the composite film decreases with increasing An-PEEK concentration in the solution. Specifically, with An-PEEK content increasing from 0 wt% to 2 wt%, 4 wt%, 8 wt% and 12 wt%, the respective CNT and An-PEEK contents within the composite films shift from 100 wt%, 0 wt% to (81.64 wt%, 18.36 wt%), (70.39 wt%, 29.61 wt%), (59.42 wt%, 40.58 wt%), and (51.47 wt%, 48.53 wt%). Fig. 3(i) presents an optical micrograph of the CNT/PEEK composite film. The thickness of CNT/PEEK composite film, measured using a digital micrometer, was $10\text{ }\mu\text{m}$. The composite film exhibits lightweight and exceptional flexibility, demonstrating tolerance to bending, curling, and repeated folding without structural failure.

Reinforcement Mechanism and Mechanical Properties

Fig. 4(a) shows the correlation between the tensile strength and elongation at break observed in CNT/PEEK-5 composite films at various mass concentrations of An-PEEK-5 infiltrated into CNT

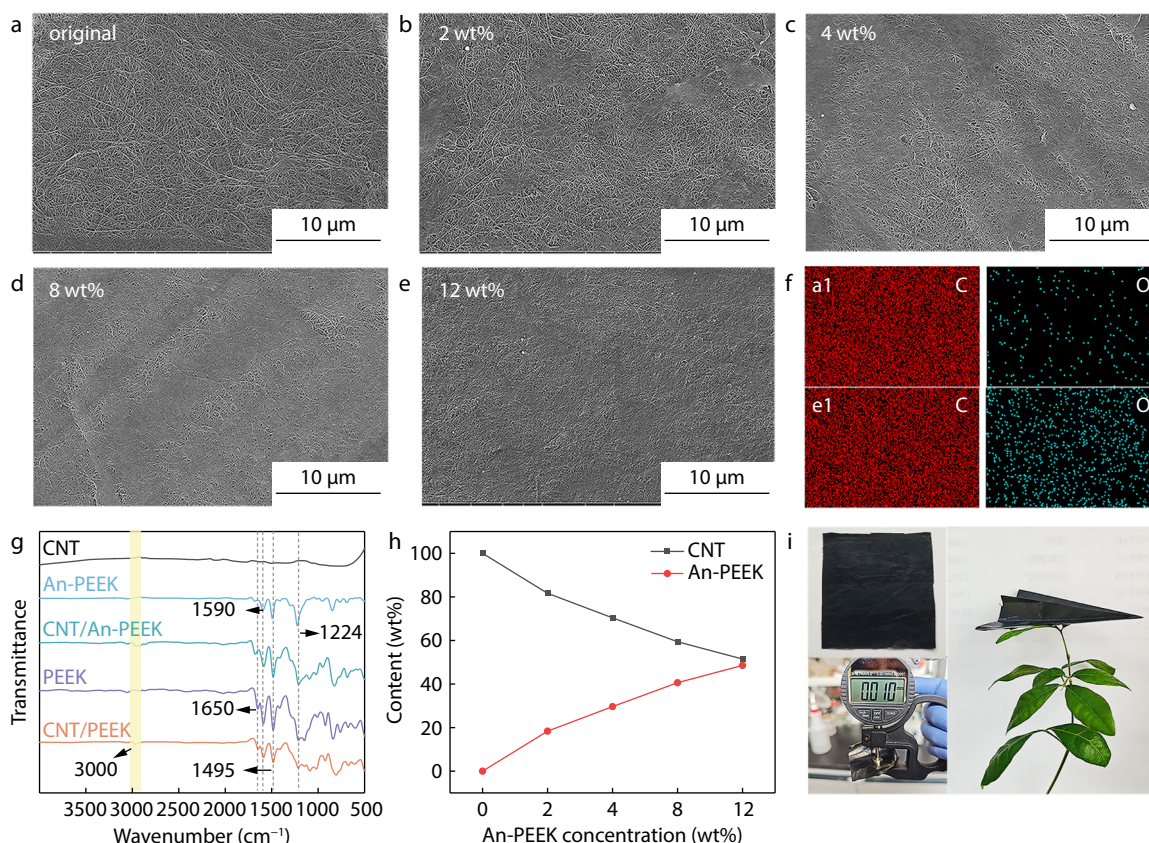


Fig. 3 (a–e) surface morphology of CNT film with different An-PEEK concentration of 0 wt%, 2 wt%, 4 wt%, 8 wt% and 12 wt%; (f) EDS analyses performed on the film surfaces (a1) 0 wt%; (e1) 12 wt%; (g) FTIR spectra of CNT, CNT/An-PEEK and CNT/PEEK films; (h) Variations of CNT and An-PEEK contents of the films prepared at different An-PEEK concentrations; (i) Macroscopic morphology, thickness and flexibility characterization of CNT/PEEK composite film.

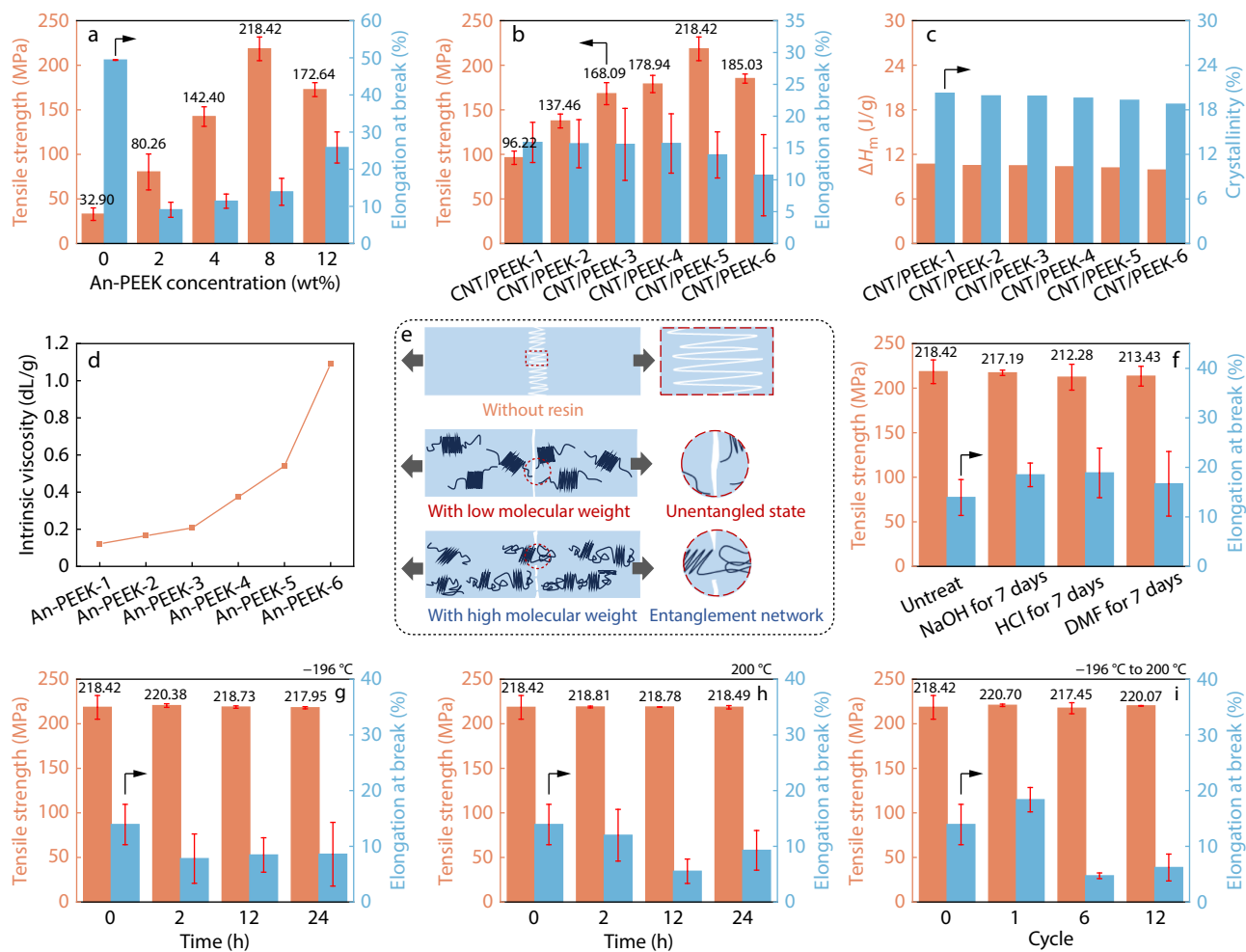


Fig. 4 Tensile properties (tensile strength and fracture elongation) of composite film across a range of An-PEEK-5 (a) mass fraction and (b) molecular weight variants; (c) ΔH_m and crystallinity of CNT/PEEK composites film; (d) Intrinsic viscosity of An-PEEK; (e) Schematic representation of the fracture mechanism at the interface phase; (f) Tensile properties of CNT/PEEK-5 composite film immersed at different chemical environments (1 mol/L HCl solution, 1 mol/L NaOH solution, and DMF) for 7 days at room temperature; (g, h) Tensile properties of CNT/PEEK composite film after treatment at 200 and -196 °C for different durations; (i) Tensile properties of CNT/PEEK composite film after 12 thermal cycles (-196 °C for 2 h and 200 °C for 2 h per cycle).

films with a constant molecular weight. Infiltration of the An-PEEK resin into the pores of the CNTs significantly improved their interconnection. Consequently, the tensile strength of the CNT/PEEK-5 composite film was substantially enhanced from 32.89 MPa (0 wt%) to 218.42 MPa (8 wt%). A decrease in tensile strength, from 218.42 MPa to 172.64 MPa, was observed for the CNT/PEEK-5 composite film upon increasing the An-PEEK mass concentration from 8 wt% to 12 wt%. These results likely stem from the excessive resin, characterized by complete surface coverage and the formation of uniform resin agglomerates. To some extent, this supersaturated resin reflected the resin properties rather than the enhanced composite properties, followed by a decrease in the tensile strength. The composite film achieved optimal mechanical properties when the resin concentration was 8 wt%.

To investigate the influence of the molecular weight of An-PEEK on the mechanical strength of the composite films, solutions containing different molecular weights (Table S1 in ESI) at 8 wt% mass concentration were prepared under identical

conditions to fabricate the corresponding films. Fig. 4(b) illustrates the tensile strength of these films as a function of the molecular weight. Specifically, the value initially rises to 218.42 MPa for CNT/PEEK-5 but subsequently declines to 185.03 MPa for CNT/PEEK-6 as the molecular weight was further increased. Analysis of the crystallinity in CNT/PEEK composite films of varying molecular weights demonstrated that the average crystallinity of the PEEK matrix remained consistently low across all samples, with minimal variation (Fig. 4c). This observation suggests that the CNT film plays two opposing roles in the polymer matrix crystallization process: it acts as a heterogeneous nucleating agent that facilitates PEEK crystallization while simultaneously serving as a physical barrier that hinders the regular arrangement of resin molecular chains, thereby suppressing crystallization. Within the processing conditions, the constraining effect dominated. Therefore, the difference in crystallinity of the composite films is not significant, and compared to the crystallinity of the pure resin, the crystallinity of the composite films decreased. With-

in the framework of this study, the crystallinity of the composite films exhibited a comparable influence on their mechanical properties; thus, it was not discussed as a dominant factor leading to performance differences.

The molecular weight fundamentally regulates the impregnation effectiveness and processability of the CNT films by determining the intrinsic viscosity of the resin solution. According to the Mark-Houwink equation: $[\eta] = KM^\alpha$ (where K and α are solvent-specific constants), $[\eta]$ is predominantly determined by the molecular weight when the chemical structure of the polymer, solvent system, and temperature are fixed. Experimental data (Fig. 4d) confirm this dependence: increasing molecular weight from 0.45×10^4 g/mol to 15.26×10^4 g/mol increases $[\eta]$ from 0.122 dL/g to 1.091 dL/g at 25 °C (the detailed viscosities are provided in Table S3 and Figs. S6–S11 in ESI). Although molecular weight enhancement leads to a steady increase in solution viscosity, the impregnation efficiency and mechanical performance of the CNT/PEEK composite films exhibit non-monotonic behavior, as evidenced by tensile testing and SEM morphological analysis (Fig. S12 in ESI). Specifically, as the molecular weight increases to 4.75×10^4 g/mol ($[\eta] = 0.541$ dL/g), the pores on the CNT film surface were gradually fully filled; only when the molecular weight exceeded this threshold, aggregation and residual pores caused by uneven resin wetting were observed. Cross-sectional SEM images of the CNT/PEEK-5 and CNT/PEEK-6 composite films are shown in Fig. S13 (in ESI). For the CNT/PEEK-5 composite film, PEEK resin can be observed throughout the interior of the CNT network, indicating the effective impregnation of the CNT framework. In contrast, the CNT/PEEK-6 composite film exhibited a relatively resin-deficient interior, whereas a larger amount of resin was retained near the film surfaces. These observations suggest that resin penetration into dense CNT networks becomes more difficult at high molecular weights. Combined with the viscosity results presented in Fig. 4(d), which demonstrate a substantial increase in solution viscosity with increasing molecular weight, the cross-sectional SEM images provide direct evidence to a certain extent that the high-viscosity An-PEEK solution exhibits restricted infiltration into the CNT film. This indicates that the molecular weight exerts a competition between viscosity-controlled infiltration kinetics and molecular-weight-dependent chain entanglement in the impregnation process. The non-monotonic trend in the mechanical characteristics of these composite films may be attributed to a competitive mechanism between two distinct effects: (A) the processing kinetic effect dominated by viscosity (high viscosity hinders the penetration of the resin into the CNT network). (B) The reinforcement and filling effect governed by the intrinsic properties of the molecular chains (longer molecular chains yield stronger entanglement, a higher density of chain entanglements, and improved mechanical strength).

Rising stage ($0.122 \text{ dL/g} < [\eta] \leq 0.541 \text{ dL/g}$): the tensile strength increases from 96.21 MPa to 218.42 MPa. Within this range, the moderate viscosity endows the solution with excellent diffusion and flow capabilities, enabling sufficient wetting and wrapping of the CNT network with the assistance of the wet pressing process. A higher molecular weight implies a larger gyration radius in the polymer chains. This enhances

the spatial coverage and bridging capability of individual polymer chains, enabling them to more effectively connect nodes within the CNT network under the premise of optimizing solution viscosity and process parameters. Such long-chain characteristics facilitate the uniform wetting of the film by the polymer solution, thereby efficiently filling the voids in the CNT network. As the molecular weight increased, the polymer chain entanglement density was significantly enhanced, which improved the intrinsic mechanical properties of the resin matrix and the interfacial stress transfer capability between the matrix and CNTs. Decreasing stage ($[\eta] > 0.541 \text{ dL/g}$): When $[\eta]$ was increased to 1.091 dL/g, the tensile strength decreases to 185.03 MPa. At this stage, the higher viscosity restricts the diffusion kinetics of the An-PEEK solution into the dense CNT network, leading to localized resin accumulation and unfilled pores on the surface (Fig. S12 in ESI). Although a higher molecular weight can enhance chain entanglement, the performance loss caused by structural defects (e.g., pores and interfacial debonding) resulting from insufficient wetting outweighs the intrinsic property gains from increased molecular weight, thereby inducing a decline in tensile strength. In summary, the tensile strength of CNT/PEEK composite films depends on the trade-off between the high chain entanglement density imparted by high molecular weight and the favorable processability guaranteed by low molecular weight. Under these solution impregnation-wet pressing process conditions, there exists an optimal molecular weight (4.75×10^4 g/mol) that achieves the best balance between intrinsic reinforcement from chain entanglement and process feasibility determined by solution viscosity, thus yielding a maximum tensile strength of 218.42 MPa. At this point, the solution viscosity is not sufficiently high to hinder adequate wetting during processing, whereas the molecular weight is sufficiently high to form a robust chain entanglement network for load bearing and transfer. Below this molecular weight, the intrinsic strength of the material is inadequate; above this value, the process fails to exert its intended efficacy, and the structural defects of the composite film dominate the failure behavior.

Before preparing CNT/PEEK composites, it is necessary to prepare CNT/An-PEEK composites, which means first synthesizing the An-PEEK resin matrix. This is because An-PEEK is soluble, which can solve the problem of PEEK being difficult to dissolve and unable to process in solution. Compared to the PEEK resin matrix, the An-PEEK resin matrix and CNT filler had similar surface energies, which facilitated the spreading and wetting of An-PEEK inside the CNT film. The specific results are as follows.

The surface energies of An-PEEK and CNT were evaluated by contact angle measurements using the OWRK method (Table S4 in ESI).^[42,43] The results show that the surface energy of An-PEEK is similar to that of the CNT film. In particular, the difference between the nonpolar and polar components was small. According to the surface energy matching theory, the smaller the difference in surface energy between two materials, the lower the interfacial energy and the better the wetting performance.^[35] Consequently, the An-PEEK resin can readily spread on and penetrate the CNT network, forming a tight interfacial contact that facilitates efficient load transfer

and contributes to the high tensile strength of the composite film. This surface-energy-driven wetting behavior is consistent with the impregnation mechanism discussed above, and further supports the optimal molecular weight selection.

The fracture surfaces of the CNT/PEEK composite films with different molecular weights are shown in Fig. S14 (in ESI). A schematic of the interfacial phase-fracture mechanism is shown in Fig. 4(e). Pristine CNT films primarily fail through inter tube sliding fractures. This failure mode arises because their porous network, stabilized by van der Waals forces and composed of loosely stacked nanotubes, exhibits a high elongation at break. However, owing to minimal interfacial bonding and poor resistance to sliding, the tensile strength remains low (Fig. S14a in ESI).^[44,45] The failure mechanism of the low-molecular-weight CNT/PEEK composite films is dominated by interfacial debonding. Short-chain PEEK molecules exhibit high mobility and tend to undergo local molecular ordering on the surface of carbon nanotubes, forming isolated and sparsely distributed ordered structures. However, because of the short length of the molecular chains, these ordered structures lack a robust entanglement network for connection, making them inherently stress concentration sites. Under external stress, cracks are prone to initiate and propagate at the boundaries of these ordered structures, leading to nanotube pull-out and interfacial debonding (Fig. S14b in ESI). In contrast, the failure mechanism of the high-molecular-weight CNT/PEEK composite films is regulated by a continuous entanglement network. Long molecular chains can span and penetrate multiple CNT bundles, forming strong and tough topological entanglement and physical crosslinking networks. This network not only effectively restricts nanotube sliding and pull-out but also dissipates substantial energy during stretching through fracture. Consequently, the composite material exhibited significantly enhanced tensile strength (Fig. S14c in ESI).^[46–48]

A stepwise curing procedure was designed to achieve near-complete impregnation and void removal from the composite film. This strategy enables the flow and redistribution of An-PEEK throughout the entire film under low temperature and pressure, thereby encapsulating CNTs, filling pores, and reducing the inter-CNT spacing. These processes are critical for enhancing the interfacial bonding and load transfer between CNTs, as well as for improving the overall homogeneity of the final film. In contrast to conventional solution impregnation (where the resin is simply cast and the solvent evaporates without external pressure), this wet-pressing process applies mechanical pressure during impregnation and curing, which not only facilitates better resin penetration but also compresses the CNT bundles, leading to a more compact and densified conductive framework that is beneficial for mechanical strength. Without wet-pressing prior to curing at 200 °C, the tensile strength was significantly reduced (Fig. S15 in ESI). From Fig. 4(f), it can be seen that the composite film still maintained its mechanical strength after corrosion treatment quite well. As depicted in Figs. 4(g)–4(i), the stability of the tensile properties of the CNT/PEEK composite films was assessed under extreme temperatures. After undergoing 24-h of high-temperature (200 °C), low-temperature (–196 °C), and 12 thermal cycles (–196 °C for 2 h and 200 °C for 2 h per cycle)

treatments, no significant degradation of the tensile strength of the composite films was observed. These results demonstrate the outstanding mechanical strength and stability of the composites in harsh environments. Measurement of the load-bearing capability of the CNT/PEEK-5 composite film, which sustains loads greater than 50000 times its mass (Fig. S16 in ESI).

EMI Shielding Performance

As shown in Fig. S17 (in ESI), the EMI SE values of the composite films with various molecular weights are nearly identical. This result clearly indicates that the impregnation process, despite using polymer solutions of different molecular weights (*i.e.*, polymer solutions of different viscosities), does not damage the preformed CNT conductive network. Therefore, the shielding performance remained unaffected, which further supports the integrity of the conductive CNT framework achieved by our fabrication method. As illustrated in Fig. 5(a), the total EMI SE (SE_T) of the CNT/PEEK composite film exhibited a distinct monotonic increase with layer accumulation. Specifically, the films attained SE_T values of 47, 54, and 64 dB for the 1-, 2-, and 3-layer configurations, respectively, within the X-band. This incremental enhancement was further corroborated by the data in Fig. 5(b), where the dominance of absorption shielding (SE_A) suggested that the multilayered architecture effectively extended the attenuation path of incident EM waves. Notably, the broadband frequency response (8.2–40 GHz) presented in Fig. 5(c) demonstrates that the composite films maintained a robust and stable shielding performance across the Ku, K, and Ka bands, confirming their reliability for broadband EMI protection. The CNT/PEEK composite film demonstrated excellent corrosion resistance, with its EMI SE maintaining its stability across diverse corrosive environments, as shown in Fig. 5(d). The stability stemmed from the semi-crystalline nature of PEEK within the CNT/PEEK composite film^[49] which imparted solvent resistance. Meanwhile, the EMI shielding performance of the CNT/PEEK composite film remained nearly unchanged after undergoing 24-h of high-temperature (200 °C), low-temperature (–196 °C), and 12 thermal cycles (–196 °C for 2 h and 200 °C for 2 h per cycle) treatments, demonstrating excellent stability under extreme temperatures (Figs. 5f–5h). High-voltage arcs produced by the Tesla coil through resonant magnetic coupling delivered sufficient energy for LED illumination.^[50] As shown in Fig. 5(e), illuminated LED bulbs located near the Tesla coil were turned off when covered by the CNT/PEEK-5 composite film, verifying its EMI SE. A control experiment was performed using a nonconductive PEEK film (Fig. S18 in ESI). Under identical conditions, the LED bulbs were illuminated.

As shown in Fig. 5(i), the EMI shielding behavior of the CNT/PEEK composite film was governed by the coupling between its conductive network and interfacial architecture. The interconnected CNT framework established efficient electron transport pathways, resulting in a pronounced impedance mismatch and initial reflection at the air-film interface.^[51] More importantly, the polymer penetrates the interfacial structure within the CNT network, generating abundant CNT-PEEK interfaces that act as polarization centers. As electromagnetic waves propagate through the film, they are progressively attenuated by conductive loss, interfacial polar-

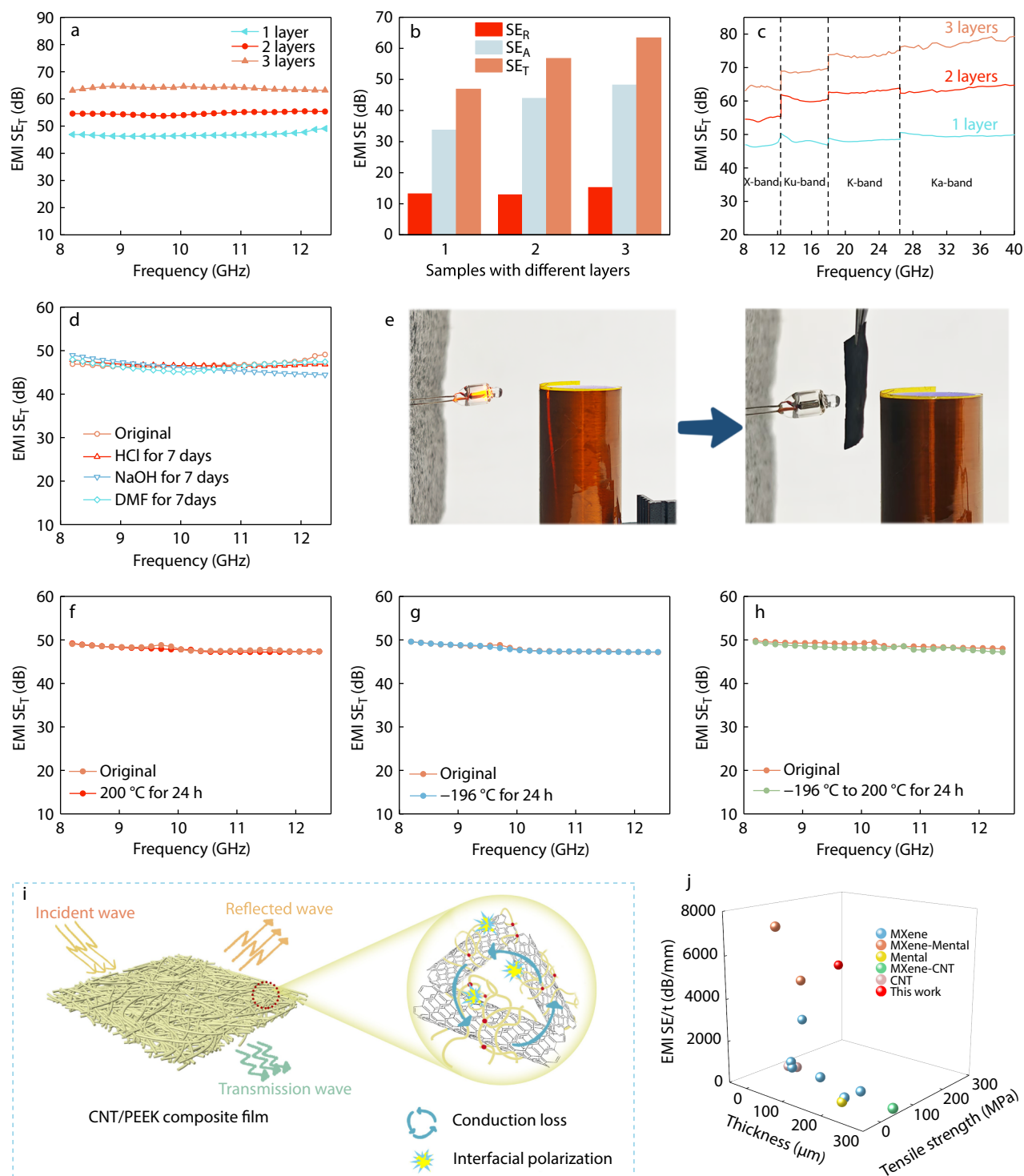


Fig. 5 EMI shielding performance of CNT/PEEK composite film. (a) SE_T and (b) SE_T , SE_R , and SE_A of single- and multi-layer CNT/PEEK composite films in the X-band; (c) SE_T of CNT/PEEK composite film with different layers across a broadband frequency range of over 8.2–40 GHz; (d) SE_T of CNT/PEEK composite film immersed in different chemical environments (1 mol/L HCl solution, 1 mol/L NaOH solution, and DMF) for 7 days at room temperature; (e) Experimental validation of EMI SE during Tesla wireless power transfer; (f, g) EMI SE of CNT/PEEK composite film after treatment at 200 and -196 °C for 24 h; (h) EMI SE of CNT/PEEK composite film after 12 thermal cycles (-196 °C for 2 h and 200 °C for 2 h per cycle); (i) Schematic of EMI shielding mechanism for CNT/PEEK composite film; (j) Comparative analysis of EMI SE/t , tensile strength and thickness for existing materials.

ization, and multiple scattering within the densified network. As shown in Fig. 5(j) and Table S5 (in ESI), the CNT/PEEK-5 composite film exhibited a high SE/t of 4700 dB/mm and a

tensile strength of 218.42 MPa. In terms of balancing the SE/t and tensile strength, the CNT/PEEK-5 composite film surpasses that of some currently published EMI materials.

Thermal Management Performance

This study investigated the photothermal and electrothermal properties as well as the extreme environmental tolerance of the CNT/PEEK composite film. Fig. 6(a) presents a schematic of the photothermal-electrothermal effect of the CNT/PEEK composite film. As shown in Fig. 6(b), the temperature variation curves of the CNT/PEEK composite film over time under different light intensities (0.3, 0.6, 1.0, 2.0 W/cm²). Under a light intensity of 2.0 W/cm², the CNT/PEEK composite film rapidly heated to approximately 178 °C and maintained a stable temperature, demonstrating outstanding photothermal conversion capability. At a lower intensity of 0.3 W/cm², the equilibrium temperature still exceeded 58 °C, indicating a retained photothermal response under weak-light conditions. Fig. 6(c) shows that as the light power density increased, the temperature of the CNT/PEEK

composite film increased and quickly stabilized. As shown in Fig. 6(d), the CNT/PEEK composite film was subjected to multiple consecutive cycles with increasing light intensities of 0.3, 1.0, and 2.0 W/cm². Under each intensity, the saturation temperature remained stable without significant attenuation after repeated-on off cycles, demonstrating excellent photothermal cycling stability across a wide power range. As shown in Fig. 6(e), the CNT/PEEK composite film exhibited negligible variation in photothermal heating profile and saturation temperature after 7-day immersion in 1 mol/L HCl, 1 mol/L NaOH, and DMF, as well as 24-h treatments at -196 °C, 200 °C, and cyclic -196 °C to 200 °C. This robust environmental stability stems from the semi-crystalline PEEK matrix: the dense crystalline domains restrict chemical diffusion, whereas the rigid crystalline framework maintains structural integrity under extreme ther-

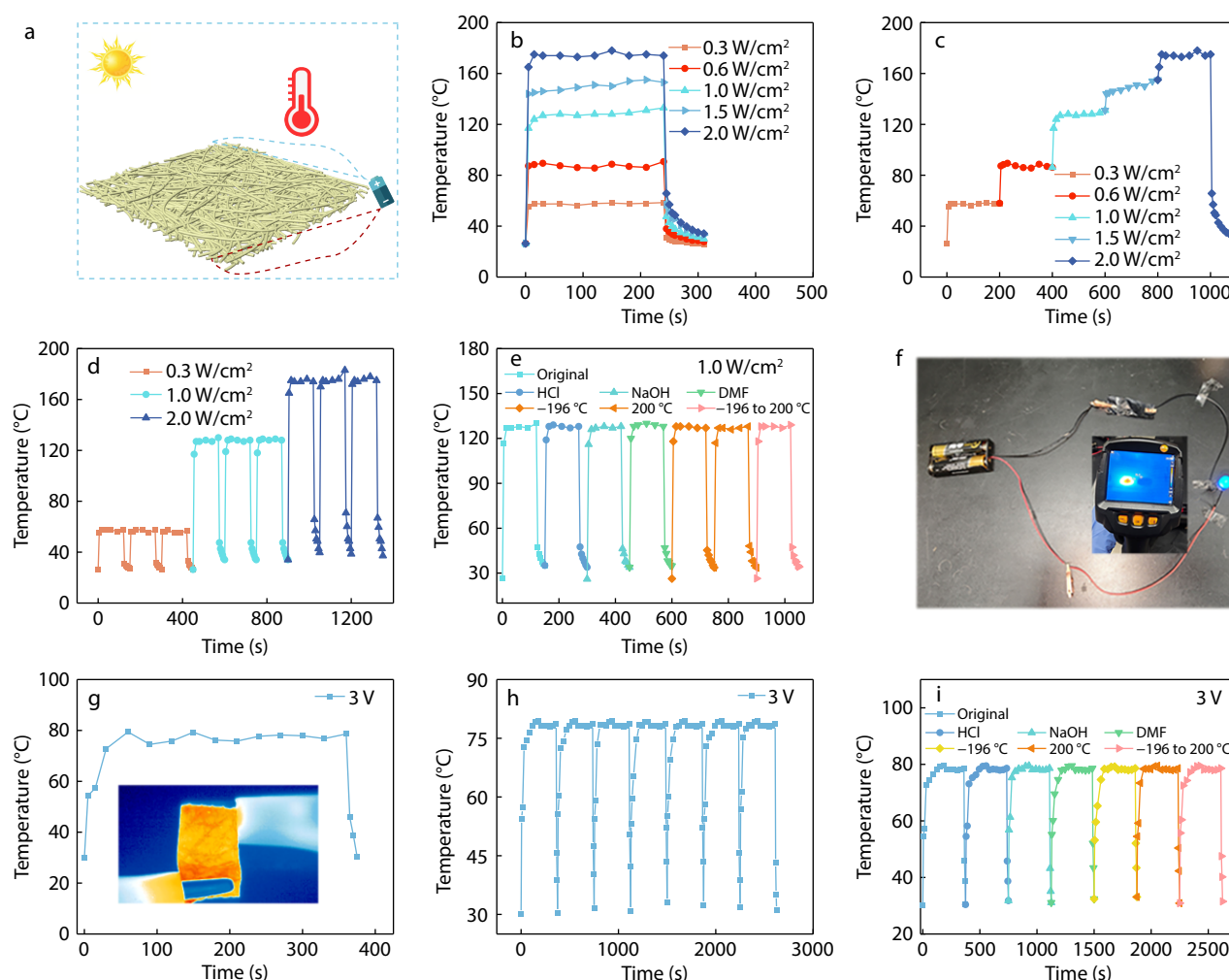


Fig. 6 (a) Schematic diagram of the photothermal-electrothermal effect of CNT/PEEK composite film; (b) Photothermal temperature-time curves of the material under different light intensities (0.3, 0.6, 1.0, 1.5, 2.0 W/cm²); (c) Photothermal temperature response curve of the material under stepwise increasing light intensity; (d) Multi-cycle photothermal stability test of the CNT/PEEK composite film under stepwise increasing light intensities of 0.3, 1.0, and 2.0 W/cm²; (e) Comparison of photothermal performance of the material under 1.0 W/cm² light irradiation, after immersion in 1 mol/L HCl solution, 1 mol/L NaOH solution, and DMF for 7 days at room temperature, as well as after extreme temperature treatments (-196 °C, 200 °C, and thermal cycling); (f) Physical device diagram for electrothermal performance test of the material; (g) Single-cycle electrothermal temperature rise curve of the material under 3 V DC voltage (inset: corresponding infrared thermal image); (h) Long-period electrothermal cycle stability test curve of the material under 3 V DC voltage; (i) Comparison of electrothermal performance of the material under 3 V DC voltage after different chemical corrosion and extreme temperature treatments.

mal conditions. Fig. 6(f) shows a physical diagram of the electrical-heating performance test. Fig. 6(g) shows the electrothermal temperature rise profile under an applied voltage of 3 V. The CNT/PEEK composite film heated rapidly to 79 °C within 60 s. The corresponding thermal image (inset) indicates excellent surface thermal uniformity. Fig. 6(h) shows the electrothermal cycling performance over seven consecutive cycles at 3 V. After prolonged on-off cycling, the steady-state temperature exhibited no attenuation, confirming the excellent electrothermal cycling stability. Fig. 6(i) compares the electrothermal response at 3 V after various treatments. Relative to the untreated sample, specimens exposed to HCl, NaOH, DMF, −196 °C, 200 °C, and thermal cycling displayed negligible variations in heating rate and equilibrium temperature. These findings verify that both the conductive network and the electrothermal conversion mechanism remain robust under extreme chemical and thermal environments. In summary, CNT/PEEK composite films that combine photothermal and electrothermal effects demonstrate considerable promise for thermal management and thermal response applications.

CONCLUSIONS

In summary, a lightweight, high-strength, and multifunctional CNT/PEEK composite film was fabricated *via* vacuum-assisted impregnation and a wet pressing process. By designing a soluble and amorphous An-PEEK precursor for crystalline PEEK resin, solution processing under mild conditions was successfully achieved, thereby overcoming the limitations of difficult solution processing of crystalline PEEK. Through the systematic modulation of the molecular weight of the PEEK matrix, a dual competition mechanism governing the impregnation behavior of polymer solutions with varying molecular weights was elucidated. The ultimate performance of the composite film was governed by a tradeoff between the processability dictated by the solution viscosity and the intrinsic reinforcement controlled by the extent of chain entanglement, rather than being determined solely by the solution viscosity. At a critical molecular weight of 4.75×10^4 g/mol ($[\eta] \approx 0.541$ dL/g), adequate fluidity for near-complete impregnation and ample chain entanglement yielded a composite film with a high tensile strength of 218.42 MPa. The highly conductive CNT film, serving as the functional filler, endowed the CNT/PEEK composite film with excellent EMI shielding performance (SE_T of 47 dB at 10 μ m, SE/t of 4700 dB/mm) and outstanding dual-conversion capabilities (electrothermal: 3 V, 79 °C; photothermal: 2.0 W/cm, 178 °C). Furthermore, the semi-crystalline nature of PEEK ensured that the composite film maintained excellent stability even after undergoing high/low-temperature impacts and long-term erosion under acid/alkali corrosive conditions. This work provides a molecular-weight-governed design strategy for high-strength polymer-based composites without a crosslinking reaction, resulting in a CNT/PEEK composite film promising for mechanical strength, EMI shielding, and thermal management in harsh environments.

Conflict of Interests

The authors declare no interest conflict.

Electronic Supplementary Information

Electronic supplementary information (ESI) is available free of charge in the online version of this article at <http://doi.org/10.1007/s10118-026-3814-3>.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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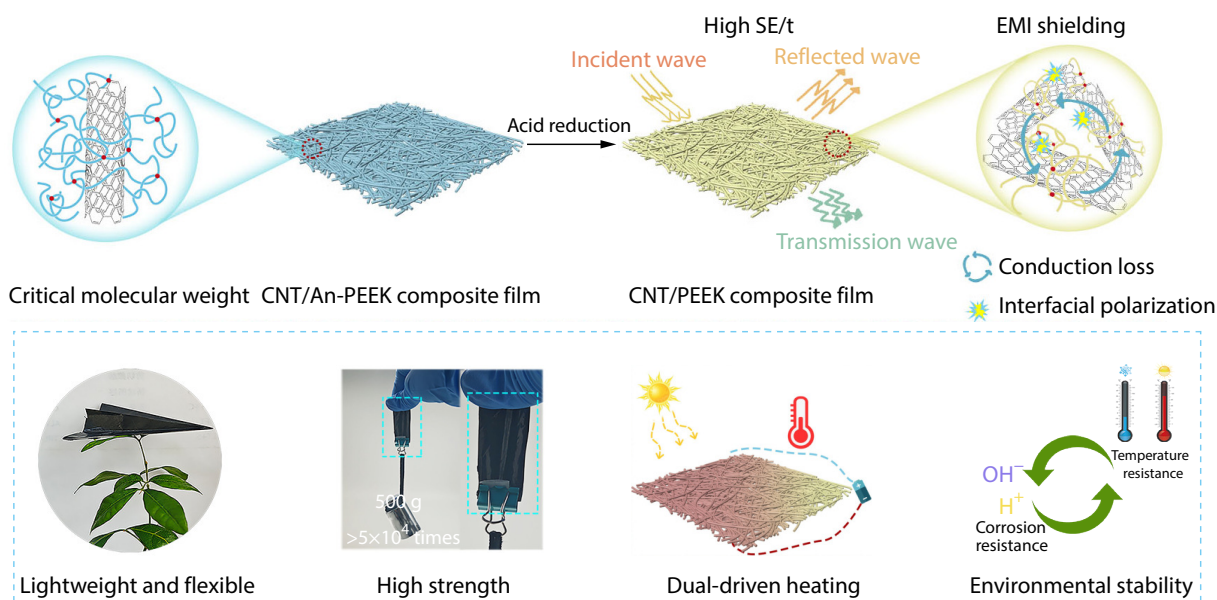
Graphical Abstract

High-strength Carbon Nanotube/Polyetheretherketone Composite Films with Tunable Impregnation for Electromagnetic Interference Shielding and Thermal Management in Harsh Environments

Chun-Qi Guo, Yu Bai, Liu-Bo Liang, Shu-Shu Jia, Xin-Yuan Shi, Gui-Bin Wang, and Shu-Ling Zhang

Jilin University

Molecular-weight regulation optimizes the balance between impregnation and chain entanglement, enabling ultrathin carbon nanotube/polyetheretherketone (CNT/PEEK) composite films with high strength, efficient electromagnetic interference (EMI) shielding, thermal management, and outstanding stability in harsh environments.



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